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Standard Guide for Examination of Hardened Concrete Using Scanning Electron Microscopy[1]

This standard is issued under the fixed designation C1723; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides information for the examination of hardened concrete using scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDX or EDS). Since the 1960s, SEM has been used for the examination of concrete and has proved to be an insightful tool for the microstructural analysis of concrete and its components. There are no standardized procedures for the SEM analysis of concrete. SEM supplements techniques of light microscopy, which are described in Practice C856/C856M, and, when applicable, techniques described in Practice C856/C856M should be consulted for SEM analysis. For further study, see the bibliography at the end of this guide.

1.2 This guide is intended to provide a general introduction to the application of SEM/EDS analytical techniques for the examination and analysis of concrete. It is meant to be useful to engineers and scientists who want to study concrete and who are familiar with, but not expert in, the operation and application of SEM/EDS technology. The guide is not intended to provide explicit instructions concerning the operation of this technology or interpretation of information obtained through SEM/EDS.

1.3 It is critical that petrographer or operator or both be familiar with the SEM/EDX (EDS) equipment, specimen preparation procedures, and the use of other appropriate procedures for this purpose. This guide does not discuss data interpretation. Proper data interpretation is best done by individuals knowledgeable about the significance and limitations of SEM/EDX (EDS) and the materials being evaluated.

1.4 The SEM provides images that can range in scale from a low magnification (for example, 15×) to a high magnification (for example, 50 000× or greater) of concrete specimens such as fragments, polished surfaces, or powders. These images can provide information indicating compositional or topographical variations in the observed specimen. The EDX (EDS) system can be used to qualitatively or quantitatively determine the elemental composition of very small volumes intersecting the surface of the observed specimen (for example, 1-10 cubic microns) and those measured compositional determinations can be correlated with specific features observed in the SEM image. See Note 1.

NOTE 1—An electronic document consisting of electron micrographs and EDX (EDS) spectra illustrating the materials, reaction products, and phenomena discussed below is available at http://netfiles.uiuc.edu/dlange/www/CML/index.html.

1.5 Performance of SEM and EDX (EDS) analyses on hardened concrete specimens can, in some cases, present unique challenges not normally encountered with other materials analyzed using the same techniques.

1.6 This guide can be used to assist a concrete petrographer in performing or interpreting SEM and EDX (EDS) analyses in a manner that maximizes the usefulness of these techniques in conducting petrographic examinations of concrete and other cementitious materials, such as mortar and stucco. For a more in-depth, comprehensive tutorial on scanning electron microscopy or the petrographic examination of concrete and concrete-related materials, the reader is directed to the additional publications referenced in the bibliography section of this guide.

1.7 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with the use of electron microscopes, X-ray spectrometers, chemicals, and equipment used to prepare samples for electron microscopy. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[1] This guide is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.65 on Petrography.

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

C125 Terminology Relating to Concrete and Concrete Aggregates

C294 Descriptive Nomenclature for Constituents of Concrete Aggregates

C295/C295M Guide for Petrographic Examination of Aggregates for Concrete

C457/C457M Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete

C856/C856M Practice for Petrographic Examination of Hardened Concrete

C1356 Test Method for Quantitative Determination of Phases in Portland Cement Clinker by Microscopical Point-Count Procedure

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *BSE, n*—backscatter electrons; these are high-energy electrons emitted back from the specimen surface. Elements of higher atomic number will have stronger emissions and appear brighter.

3.1.2 *brightness, n*—the amount of energy used to produce an X-ray.

3.1.3 *charging, n*—the buildup of electrons on the specimen at the point where the beam impacts the sample. Charging can alter the normal contrast of the image (usually becomes brighter) and may deflect the beam. Coating the specimen with a thin layer of conductive material (such as gold or carbon) can minimize this effect.

3.1.4 *contrast, n*—the difference in intensity of the energy produced by varying elements when excited.

3.1.5 *dead-time, n*—the time of finite processing during which the circuit is "dead" and unable to accept a new pulse from the X-rays.

3.1.6 *EDX (EDS) (energy-dispersive X-ray spectroscopy), n*—the interaction of the electron beam with atoms in the sample produces characteristic X-rays having energies and wavelengths unique to atoms.

3.1.7 *live-time, n*—how the acquistion of X-ray data is timed when the rate of X-ray events between measurements are compared. Opposite of dead-time.

3.1.8 *K, L, or M peaks, n*—characteristic X-ray intensities detected for elements.

3.1.9 *raster, n*—to scan as when the beam from the filament sweeps back and forth over the sample

3.1.10 *SE, n*—secondary electrons; these are low-energy electrons emitted when the specimen is hit with the beam and associated with the topography of the same.

3.1.11 *SEM, n*—scanning electron microscope.

3.1.12 *stage, n*—platform upon which the specimen is placed within the vacuum chamber that can be remotely moved in various directions.

3.1.13 *working distance, n*—the distance between the detector and the sample. Each SEM will have an optimun distance in which X-rays can be collected for EDX (EDS).

3.1.14 *X-ray detector, n*—also known as EDX (EDS) system.

4. Description of Equipment

4.1 The principles of the electron system of the scanning electron microscope, the interactions of the electron beam and the specimen under examination, and the detection systems used for the examination are based on concepts that need understanding if the resulting image and other analytical information obtained are to be best resolved and understood. An abbreviated discussion is provided here. A more comprehensive understanding can be obtained from texts devoted to this subject (**1**,**2**).[3]

4.1.1 *SEM Optics:*

4.1.1.1 An electron beam is generated in a column consisting of an electron gun and multiple electromagnetic lenses and apertures. The electron beam is generated by heating a filament so that it emits electrons. The most common filament for general SEM work is tungsten, but other filaments can be used for increased brightness. The electrons are accelerated towards the specimen by an applied potential and then focused by lenses and apertures. The energy of the electron beam influences resolution, image quality, and quantitative and qualitative X-ray microanalyses.

4.1.1.2 The electron beam is finely focused through electromagnetic lenses and apertures. A smaller beam size improves resolution, but decreases signal intensity.

4.1.1.3 Electron systems operate under vacuum. Specimens should be prepared to minimize alteration or damage when they are exposed to the vacuum (See 5.1.4). Variable pressure scanning electron microscopes, low vacuum scanning electron microscopes (LVSEM), and environmental scanning electron microscopes (ESEM) permit the examination of samples containing some moisture under low vacuum. The ESEM also allows analysis of organic materials. Even in an ESEM, however, some drying occurs.

4.1.2 *Signal Generation and Detection:*

4.1.2.1 The interaction of the electron beam with the sample generates several types of signals that can be utilized for imaging and X-ray microanalysis. The intensities of these signals are measured by detectors. The signals allow the examination and determination of properties such as surface topography, elemental composition, and spatial distribution of components. Signal intensities are generally used to provide an image on a screen.

4.1.2.2 The signals that are produced when the electron beam strikes the specimen surface are secondary electrons (SE), backscattered electrons (BSE), and X-rays.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.